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THERMOPLASTIC FIBERS AND FABRICS

Abstract:

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Fibers comprising one or more thermoplastic hydroxy-functionalized polyethers or polyesters, prepared by the reaction of a dinucleophilic monomer with a diglycidyl ether, a diglycidyl ester or epihalohydrin and, optionally a polymer which is not a hydroxy-functionalized polyether or polyester, including polyolefin, polyester, polyamide, polysaccharide, modified polysaccharide or naturally-occurring fiber or particulate filler; thermoplastic polyurethane, thermoplastic elastomer or glycol-modified copolyester. Data supplied from the esp@cenet database - Worldwide

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(54) Title: THERMOPLASTIC FIBERS AND FABRICS

(57) Abstract

Fibers comprising one or more thermoplastic hydroxy-functionalized polyethers or polyesters, prepared by the reaction of a dinucleophilic monomer with a diglycidyl ether, a diglycidyl ester or epihalohydrin and, optionally a polymer which is not a hydroxy-functionalized polyether or polyester, including polyolefin, polyester, polyamide, polysaccharide, modified polysaccharide or naturally-occurring fiber or particulate filler; thermoplastic polyurethane, thermoplastic elastomer or glycol-modified copolyester.

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THERMOPLASTIC FIBERS AND FABRICS

The present invention relates to thermoplastic fibers and fabrics.

It is known to prepare fibers, yarns and fabrics from polystyrene, vinyl polymers, nylons, polyesters, polyolefins, or fluorocarbons. See, for example, 5 U.S. Patents 4,181,762; 4,945,150; 4,909,975 and 5,071,917.

However, it still remains desirable to provide fibers prepared from polymers which have not been used as starting materials for the preparation of fibers, 10 yarns and fabrics. These fibers have exceptional properties with respect to bonding, hydrophilicity and chemical resistance which is a special feature of epoxy-based polymers.

In a first aspect, the present invention is a fiber comprising at least one thermoplastic hydroxy-functionalized polyether or polyester and, optionally, a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

In a second aspect, the present invention is a bicomponent fiber having (1) 15 a first component comprising a thermoplastic hydroxy-functionalized polyether or polyester or a blend of hydroxy-functionalized polyether or polyester and (2) a second component comprising a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

In a third aspect, the present invention is a method of forming a non- 20 woven fabric by forming a web of at least one fibrous component and heating the web to cause bonding of fibrous components of the web, characterized in that at least one fibrous component comprises a thermoplastic hydroxy-functionalized polyether or polyester.

The fiber of the present invention can be a single component or a 25 bicomponent fiber.

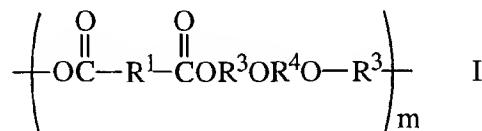
The single component fiber comprises at least one thermoplastic hydroxy-functionalized polyether or polyester and, optionally, a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

The bicomponent fiber of the present invention has (1) a first component 30 comprising a thermoplastic hydroxy-functionalized polyether or polyester or a blend of hydroxy-functionalized polyether or polyester and (2) a second component comprising a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

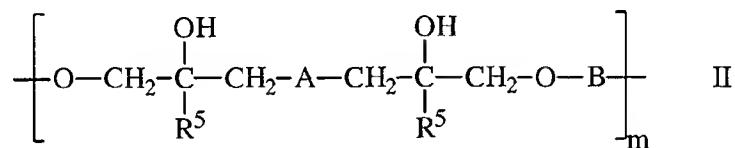
In general, the thermoplastic hydroxy-functionalized polyethers or polyesters are prepared by the reaction of a dinucleophilic monomer with a diglycidyl ether, a diglycidyl ester or epihalohydrin.

Preferably, the thermoplastic hydroxy-functionalized polyether or polyester
5 is selected from :

(1) poly(hydroxy ester ethers) or poly(hydroxy esters) having repeating units represented by the formula:

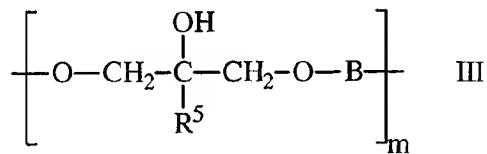


(2) polyetheramines having repeating units represented by the formula:



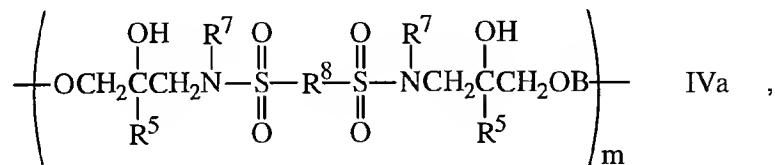
10

(3) hydroxy-functionalized polyethers having repeating units represented by the formula:

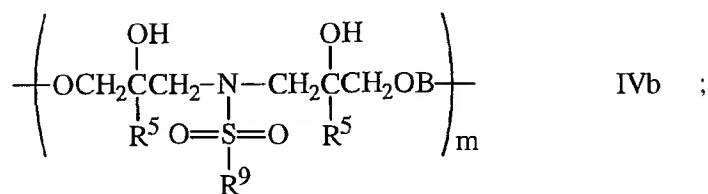


or

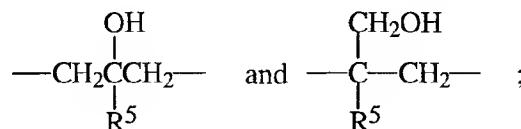
(4) hydroxy-functionalized poly(ether sulfonamides) having repeating units
15 represented by the formula:



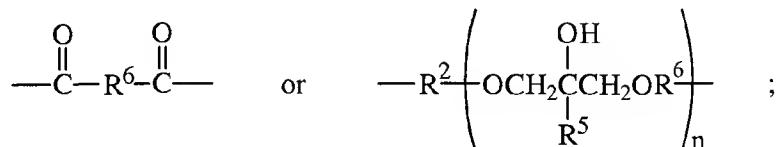
or



wherein R¹ is a divalent organic moiety which is primarily hydrocarbon; R² is independently a divalent organic moiety which is primarily hydrocarbon; R³ is



R⁴ is



5

R⁵ is hydrogen or alkyl; R⁶ is a divalent organic moiety which is primarily hydrocarbon; R⁷ and R⁹ are independently alkyl, substituted alkyl, aryl, substituted aryl; R⁸ is a divalent organic moiety which is primarily hydrocarbon; A is an amine moiety or a combination of different amine moieties; B is a divalent organic moiety which is primarily hydrocarbon; m is an integer from 5 to 1000; and n is an integer from 0 to 100.

10 In the preferred embodiment of the present invention, A is 2-hydroxyethylimino-, 2-hydroxypropyl-imino-, piperazinyl, N,N'-bis(2-hydroxyethyl)-1,2-ethylenedimino; and B and R¹ are independently 1,3-phenylene, 1,4-phenylene; sulfonyldiphenylene, oxydiphenylene, thiodiphenylene or isopropylidene-diphenylene; R⁵ is hydrogen; R⁷ and R⁹ are independently methyl, ethyl, propyl, butyl, 2-hydroxyethyl or phenyl; and B and R⁸ are independently 1,3-phenylene, 1,4-phenylene, sulfonyldiphenylene, oxydiphenylene, thiodiphenylene or isopropylidenediphenylene.

15 The poly(hydroxy ester ethers) represented by Formula I are prepared by reacting diglycidyl esters of aliphatic or aromatic diacids, such as diglycidyl terephthalate, or diglycidyl ethers of dihydric phenols with aliphatic or aromatic diacids such as adipic acid or isophthalic acid. These polyesters are described in U.S. Patent 5,171,820. Alternatively, the poly(hydroxyester ethers) are prepared by reacting a diglycidyl ester with a bisphenol or by reacting a diglycidyl ester, diglycidyl ether, or an epihalohydrin with a dicarboxylic acid.

20 The polyetheramines represented by Formula II also referred to as poly(hydroxy amino ethers) are prepared by contacting one or more of the diglycidyl ethers of a dihydric phenol with an amine having two amine hydrogens under conditions sufficient to cause the amine moieties to react with epoxy moieties to form a polymer backbone having amine linkages, ether linkages and pendant hydroxyl moieties. These

polyetheramines are described in U.S. Patent 5,275,853. The polyetheramines can also be prepared by contacting a diglycidyl ether or an epihalohydrin with a difunctional amine.

The hydroxy-functionalized polyethers represented by Formula III are prepared, for example, by contacting a diglycidyl ether or a combination of diglycidyl ethers with a dihydric phenol or combination of dihydric phenols using the process described in U.S. Patent 5,164,472. Alternatively, the poly(hydroxy ethers) are obtained by allowing a dihydric phenol or a combination of dihydric phenols to react with an epihalohydrin by the process described by Reinking, Barnabeo, and Hale in the *Journal of Applied Polymer Science, Volume 7, page 2135 (1963)*.

1.0 The hydroxy-functionalized poly(ether sulfonamides) represented by Formulae IVa and IVb are prepared, for example, by polymerizing an N,N'-dialkyl or N,N'-diaryldisulfonamide with a diglycidyl ether as described in U.S. Patent 5,149,768.

The hydroxy-functionalized polyethers commercially available from Phenoxy Associates, Inc. are also suitable for use in the present invention. These hydroxy-functionalized polyethers are the condensation reaction products of a dihydric polynuclear phenol, such as bisphenol A, and an epihalohydrin and have the repeating units represented by Formula III wherein B is an isopropylidene diphenylene moiety. These hydroxy-phenoxyether polymers and the process for preparing them are described in U.S. Patents 3,305,528. Other hydroxy functional polyethers that are suitable for use in the present invention are poly(alkylene oxides), which are typically produced through the polymerization of ethylene oxide, propylene oxide or butylene oxide. Specific examples include, but are not limited to, poly(ethylene oxide), poly(propylene oxide), poly(butylene oxide), or copolymers containing varying amounts of different poly(alkylene oxides). These polymers also may be particularly suitable for blending with polymers of any of Formulas I through IV. Advantages of blends of poly(alkylene oxides) and polymers of Formulas I through IV include the ability to manipulate the glass transition temperature of the blends or to modify hydrophilicity.

The polymers which are not hydroxy-functionalized polyesters or polyethers which can be employed in the practice of the present invention for preparing the fibers include polyolefins, polyesters, polyamides, polysaccharides, modified polysaccharides or naturally-occurring fibers or particulate fillers; thermoplastic polyurethanes, thermoplastic elastomers and glycol-modified copolyester (PETG). Other polymers of the polyester or polyamide type can also be employed in the practice of the present invention for preparing the fiber. Such polymers include poly(hexamethylene adipamide), polycaprolactone, poly(hexamethylene sebacamide), poly(ethylene 2,6-

naphthalate) and poly(ethylene 1,5-naphthalate), poly(tetramethylene 1,2-dioxybenzoate) and copolymers of ethylene terephthalate and ethylene isophthalate.

The polyesters and methods for their preparation are well known in the art and reference is made thereto for the purposes of this invention. For purposes of 5 illustration and not limitation, reference is particularly made to pages 1-62 of Volume 12 of the *Encyclopedia of Polymer Science and Engineering*, 1988 revision, John Wiley & Sons.

The polymers which are not hydroxy-functionalized polyesters or polyethers can be blended with the hydroxy-functionalized polyether or polyester at levels 10 of less than 50 weight percent and, preferably less than 30 weight percent, based on the weight of the fiber. These other polymers can be blended into the hydroxy-functionalized polyether or polyester in order to reduce composition cost, to modify physical properties, barrier or permeability properties, or adhesion characteristics. In the case of bicomponent fibers, the separate non-hydroxy-functional-containing component may be 15 used at levels of up to 99 percent, preferably less than 95 percent, based on the weight of the fiber.

The polyamides which can be employed in the practice of the present invention for preparing the fibers include the various grades of nylon, such as nylon 6, nylon 6,6 and nylon 12.

20 By the term "polyolefin" is meant a polymer or copolymer derived from simple olefin monomers such as ethylene, propylene, butylene, or isoprene, and one or more monomers copolymerizable therewith. Such polymers (including raw materials, their proportions, polymerization temperatures, catalysts and other conditions) are well-known in the art and reference is made thereto for the purpose of this invention.

25 Additional comonomers which can be polymerized with ethylene include olefin monomers having from 3 to 12 carbon atoms, ethylenically unsaturated carboxylic acids (both mono- and difunctional) and derivatives of such acids such as esters (for example, alkyl acrylates) and anhydrides; monovinylidene aromatics and monovinylidene aromatics substituted with a moiety other than halogen such as styrene and methylstyrene; and 30 carbon monoxide. Exemplary monomers which can be polymerized with ethylene include 1-octene, acrylic acid, methacrylic acid, vinyl acetate and maleic anhydride.

The polyolefins which can be employed in the practice of the present invention for preparing the fibers include polypropylene, polyethylene, and copolymers and blends thereof, as well as ethylene-propylene-diene terpolymers. Preferred

polyolefins are polypropylene, such as Pro-fax™ PF635 (Trademark of Montell North American Inc.) and INSPIRE™ (Trademark of The Dow Chemical Company), linear high density polyethylene (HDPE), heterogeneously-branched linear low density polyethylene (LLDPE) such as DOWLEX™ polyethylene resin (Trademark of The Dow Chemical Company), heterogeneously-branched ultra low linear density polyethylene (ULDPE) such as ATTANE™ ULDPE (Trademark of The Dow Chemical Company); homogeneously-branched, linear ethylene/α-olefin copolymers such as Tafmer™ (Trademark of Mitsui Petrochemicals Company Limited) and Exact™ (Trademark of Exxon Chemical Company); homogeneously branched, substantially linear ethylene/α-olefin polymers such as AFFINITY™ (Trademark of The Dow Chemical Company) and ENGAGE® (Trademark of DuPont Dow Elastomers L.L. C) polyolefin elastomers, which can be prepared as disclosed in U.S. Patents 5,272,236 and 5,278,272; and high pressure, free radical polymerized ethylene polymers and copolymers such as low density polyethylene (LDPE), ethylene-acrylic acid (EAA) copolymers such as PRIMACOR™ (Trademark of The Dow Chemical Company), and ethylene-vinyl acetate (EVA) copolymers such as Escorene™ polymers (Trademark of Exxon Chemical Company), and Elvax™ (Trademark of E.I. du Pont de Nemours & Co.). The more preferred polyolefins are the homogeneously-branched linear and substantially linear ethylene copolymers with a density (measured in accordance with ASTM D-792) of 0.85 to 0.99 g/cm³, a weight average molecular weight to number average molecular weight ratio (Mw/Mn) from 1.5 to 3.0, a measured melt index (measured in accordance with ASTM D-1238 (190/2.16)) of 0.01 to 100 g/10 minutes, and an I10/I2 of 6 to 20 (measured in accordance with ASTM D-1238 (190/10)).

In general, high density polyethylene (HDPE) has a density of at least about 0.94 grams per cubic centimeter (g/cc) (ASTM Test Method D-1505). HDPE is commonly produced using techniques similar to the preparation of linear low density polyethylenes. Such techniques are described in U.S. Patents 2,825,721; 2,993,876; 3,250,825 and 4,204,050. The preferred HDPE employed in the practice of the present invention has a density of from 0.94 to 0.99 g/cc and a melt index of from 0.01 to 35 grams per 10 minutes as determined by ASTM Test Method D-1238.

The polysaccharides which can be employed in the practice of the present invention are the different starches, celluloses, hemicelluloses, xylanes, gums, pectins and pullulans. Polysaccharides are known and are described, for example, in *Encyclopedia of Polymer Science and Technology, 2nd edition, 1987*. The preferred polysaccharides are starch and cellulose.

The modified polysaccharides which can be employed in the practice of the present invention are the esters and ethers of polysaccharides, such as, for example, cellulose ethers and cellulose esters, or starch esters and starch ethers. Modified polysaccharides are known and are described, for example, in *Encyclopedia of Polymer Science and Technology, 2nd edition, 1987.*

The term "starch" as used herein, refers to carbohydrates of natural vegetable origin, composed mainly of amylose and/or amylopectin, and includes unmodified starches, starches which have been dewatered but not dried, physically modified starches, such as thermoplastic, gelatinized or cooked starches, starches with a modified acid value (pH) where acid has been added to lower the acid value of a starch to a range of from 3 to 6, gelatinized starches, ungelatinized starches, cross-linked starches and disrupted starches (starches which are not in particulate form). The starches can be in granular, particulate or powder form. They can be extracted from various plants, such as, for example, potatoes, rice, tapioca, corn, pea, and cereals such as rye, oats, and wheat.

Celluloses are known and are described, for example, in *Encyclopedia of Polymer Science and Technology, 2nd edition, 1987.* Celluloses are natural carbohydrate high polymers (polysaccharides) consisting of anhydroglucose units joined by an oxygen linkage to form long molecular chains that are essentially linear. Cellulose can be hydrolyzed to form glucose. The degree of polymerization ranges from 1000 for wood pulp to 3500 for cotton fiber, giving a molecular weight of from 160,000 to 560,000. Cellulose can be extracted from vegetable tissues (wood, grass, and cotton). Celluloses can be used in the form of fibers.

The naturally-occurring fibers or particulate fillers which can be employed in the practice of the present invention are, for example, wood flour, wood pulp, wood fibers, cotton, flax, hemp, or ramie fibers, rice or wheat straw, chitin, chitosan, cellulose materials derived from agricultural products, nut shell flour, corn cob flour, and mixtures thereof.

In general, the fibers of the present invention can be formed by well known processes such as melt spinning, wet spinning, or conjugate spinning. The fibers of the present invention may be extruded into any size, or length desired. They may also be extruded into any shape desired, such as, for example, cylindrical, cross-shaped, trilobal or ribbon-like cross-section.

The bicomponent fibers of the present invention can have the following fiber cross-section structures:

- (1) Side-by-side
- (2) Sheath-core
- 5 (3) Islands-in-the sea and
- (4) Citrus (Segmented pie)

(1) Side-by-side

A method for producing side-by-side bicomponent fibers is described in U.S. Patent 5,093,061. The method comprises (1) feeding two polymer streams through 10 orifices separately and converging at substantially the same speed to merge side-by-side as a combined stream below the face of the spinneret; or (2) feeding two polymer streams separately through orifices, which converge at the surface of the spinneret, at substantially the same speed to merge side-by-side as a combined stream at the surface of the spinneret. In both cases, the velocity of each polymer stream at the point of merge 15 is determined by its metering pump speed and the size of the orifice. The fiber cross-section has a straight interface between two components.

Side-by-side fibers are generally used to produce self-crimping fibers. All commercially available self-crimping fibers are produced by using a system based on the different shrinkage characteristics of each component.

20 (2) Sheath-core

Sheath-core bicomponent fibers are those fibers where one of the components (core) is fully surrounded by a second component (sheath). Adhesion is not always essential for fiber integrity.

The most common way to produce sheath-core fibers is a technique in 25 which two polymer liquids (melts) are separately led to a position very close to the spinneret orifices and then extruded in sheath-core form. In the case of concentric fibers, the orifice supplying the "core" polymer is in the center of the spinning orifice outlet and flow conditions of core polymer fluid are strictly controlled to maintain the concentricity of both components when spinning. Modifications in spinneret orifices enable one to obtain 30 different shapes of core or/and sheath within the fiber cross-section.

The sheath-core structure is employed when it is desirable for the surface to have the property of one of the polymers such as luster, dyeability or stability, while the

core may contribute to strength, and reduced cost. The sheath-core fibers are used as crimping fibers and as bonding fibers in the non-woven industry.

The sheath-core bicomponent fiber can have a core comprising the hydroxy-functionalized polyether or polyester and a sheath comprising a polymer which is not a hydroxy-functionalized polyether or polyester. Alternatively, the hydroxy-functionalized polyether or polyester can be the sheath and the polymer that is not the hydroxy-functionalized polyether or polyester the core of the bicomponent fiber. The sheath-core may be circular in cross-section or may have some other geometry, such as trilobal. A variant such as "tipped trilobal" can also be constructed wherein the sheath component is no longer continuous about the core but exists only at the tips of the lobes formed by the core. Other configurations which may be utilized are illustrated in *International Fiber Journal, Volume 13, No. 3, June 1998* in the articles beginning on pages 20, 26, and 49.

Methods for producing sheath - core bicomponent fibers are described in 15 U.S. Patents 3,315,021 and 3,316,336.

(3) Islands-in the-sea

Islands-in-the sea fibers are also called matrix-filament fibers which include heterogeneous bicomponent fibers. A method for producing islands-in-the sea fibers is described in U.S. Patent 4,445,833. The method comprises injecting streams of 20 core polymer into sheath polymer streams through small tubes with one tube for each core stream. The combined sheath-core streams converge inside the spinneret hole and form one island-in-the sea conjugate stream.

Mixing the different polymer streams with a static mixer in the spinning process also makes island-in-the-sea bicomponent fibers. The static mixer divides and 25 redivides the polymer stream to form a matrix stream with multiple cores. This method for producing island-in-the-sea fibers is described in U.S. Patent 4,414,276.

The hydroxy-functionalized polyether or polyester can be the sea polymer and the polymer which is not a hydroxy-functionalized polyether or polyester can be the island polymer. The hydroxy-functionalized polyether or polyester can also be the island 30 polymer and the polymer which is not a hydroxy-functionalized polyether or polyester, the sea polymer.

The islands-in-the-sea structure is employed when it is desirable to increase the modulus of the fiber, reduce moisture regain, reduce dyeability, improve the texturing capability or give the fiber a unique lustrous appearance.

(4) Citrus type (Segmented pie)

5 The citrus type bicomponent or segmented pie bicomponent fibers can be made by polymer distribution and/or spinneret modifications of the pack assemblies employed in the methods described above for producing the side-by-side, sheath-core or islands-in-the-sea fibers. For example, by introducing a first polymer stream and a second polymer stream alternately through eight radial channels toward the spinneret 10 hole instead of two channels, the resultant fiber is an eight-segment citrus type fiber. If the spinneret orifice has the configuration of three or four slots on a circle (a common orifice configuration to produce hollow fibers), the fiber is a hollow citrus type fiber with eight segments. The hollow citrus type fiber can also be made by the use of special spinneret orifice configurations with a sheath-core spin pack as described in U.S. Patents 15 4,246,219 and 4,357,290.

The fibers of the present invention can be blended with other synthetic or natural fibers, such as carbon fibers, cotton, wool, polyester, polyolefin, nylon, rayon, glass fibers, fibers of silica, silica alumina, potassium titanate, silicone carbide, silicone nitride, boron nitride, boron, acrylic fibers, tetrafluoroethylene fibers, polyamide fibers, 20 vinyl fibers, protein fibers, ceramic fibers, such as aluminum silicate, and oxide fibers, such as boron oxide.

Additives such as pigments, stabilizers, impact modifiers, plasticizers, carbon black, conductive metal particles, abrasives and lubricating polymers may be incorporated into the fibers. The method of incorporating the additives is not critical. The 25 additives can conveniently be added to the hydroxy-functionalized polyether or polyester prior to preparing the fibers. If the hydroxy-functionalized polyether or polyester is prepared in solid form, the additives can be added to the melt prior to preparing the fibers.

The fibers of the present invention can be crosslinked by chemical 30 treatment, heating or irradiation with ultraviolet light. For example, the fibers can be chemically treated with crosslinking agents such as diisocyanates, glycidylmethacrylate, bisepoxides and anhydrides.

The fibers of the present invention are suitable for use in filtration media, binder fibers for glass or carbon fibers, binder fibers in non-woven fabrics made of

thermoplastic polymers which are not hydroxy-functionalized polyethers or polyesters or binder fibers in non-woven fabrics made of cellulosic-based materials. These fibers are also useful in the manufacture of medical apparels. They are also useful in making woven and non-woven fabrics which can be used in making apparels, water-absorbent 5 cloths, antistatic wipes, or water-absorbent mats.

Woven fabrics are formed from the fibers of the present invention by techniques commonly used in the woven textiles industry, such as weaving or knitting.

Non-woven fabrics are based on a fibrous web. The fibers of the present invention can be formed into webs using the following known technologies:

- 10 (1) Dry-formed, carded or air-laid and bonded - The webs are formed from staple fibers by carding or air-laying, bonded overall or in a pattern with latex or other water-borne adhesives. In carding, clumps of staple fibers are separated mechanically into individual fibers and formed into a coherent web. In air-laying, fibers are introduced into an airstream and are captured on a screen from the air 15 stream.
- (2) Thermal-bonded - Dry-formed webs of staple fibers are bonded with fusible fibers or composed entirely of fusible fibers.
- (3) Air-laid - Wood pulp fibers, with or without added staple fibers, are bonded with latex or similar adhesives.
- 20 (4) Wet-formed - Short fibers are formed into a web by processes derived from paper-making technology, followed by bonding with latex or thermal binders.
- (5) Spun-bonded - Webs composed of long filaments with normal textile diameter are formed directly from bulk polymer and are usually bonded thermally.
- (6) Melt-blown - Webs of long, extremely fine diameter fibers are formed directly 25 from bulk polymer and are usually bonded by hot embossing processes.
- (7) Spun-laced - Dry-formed webs are mechanically entangled by multiple fine, high pressure water jets, in most cases, without adhesive binder.
- (8) Needle-punched - Fibers are mechanically entangled by multiple reciprocating banks of barbed needles.
- 30 (9) Laminated - Different layers are combined in composite or reinforced fabric by an adhesive, thermal fusion or entanglement.

(10) Stitch-bonded - Staple fiber webs are reinforced or mechanically entrapped by yarns stitched or knitted through the webs.

Non-woven fabrics and processes for preparing them are described in the *Encyclopedia of Polymer Science And Engineering*, Second Edition, Volume 10, 5 pages 204-251.

The following working examples are given to illustrate the invention and should not be construed as limiting its scope. Unless otherwise indicated, all parts and percentages are by volume.

Example 1

10 A 3/8-inch (0.95 cm) single-screw extruder fitted with an 8-hole spinneret was used to spin monofilaments based on 100 percent thermoplastic hydroxy-functional polyether. The melt temperature was 200°C. The fibers were rolled onto bobbins without further stretching.

Example 2

15 Bicomponent fibers containing polypropylene as the core and a thermoplastic hydroxy-functionalized polyether as the sheath were spun. Two single-screw extruders, one feeding the polypropylene and another feeding the thermoplastic hydroxy-functionalized polyether were used. The extruders fed the molten polymers (melt temperature was 200°C) to a 288-holed spinneret from which the bicomponent 20 fibers were spun. Fibers with polypropylene:thermoplastic hydroxy-functionalized polyether ratios of 90:10, 80:20, 70:30, 40:60 and 50:50 were spun. The fibers were also stretched after exiting the spinneret by using extender rolls and then taken up on bobbins.

Example 3

25 A polyetheramine poly(hydroxy amino ether) (derived from the reaction of the diglycidyl ether of bisphenol A and ethanolamine) and a polypropylene were spun into bicomponent fibers. The poly(hydroxy amino ether) had an MFI (Melt Flow Index) of 8 at 230°C using a 2.16 kg weight. The polypropylene source was a 35 MFI Pro-fax™ PF635 30 polypropylene from Montell. This sheath/core bicomponent fiber was produced under the conditions listed in Table I.

Table I

<u>GENERAL</u>	<u>SHEATH EXTRUDER</u>	<u>CORE EXTRUDER</u>
Polymer Type	Dow Polyetheramine (8 MFI)	Polypropylene (35 MFI)
Ratio (w/w)	20	80
Meter Pump (rpm)	6.46	39.45
<u>Extrusion Temps,</u> (°C)		
Zone 1	185	210
Zone 2	200	220
Zone 3	200	220
Zone 4	210	220
Spin Head	210	210
Pack Pressure (psi)	2600	1900

<u>ROLL CONDITIONS</u>	<u>SPEED (METERS/MINUTE)</u>	<u>TEMP (°C)</u>
Denier Roll	1500	Ambient
Tension Roll	1500	Ambient
Draw Roll # 1	1400	50°C
Draw Roll # 2	1575	50

5

Example 4

A 30/70 weight/weight (w/w) poly(hydroxy amino ether) (derived from the reaction of the diglycidyl ether of bisphenol A and ethanolamine) sheath/polypropylene core bicomponent fiber was treated on a Tech Tex texturizing unit to impart a crimp. This equipment uses the stuffer box method of texturizing. The texturized yarn was cut into staple fiber of 2-inch (5 cm) length on an Ace Strip Cutter, Model C-75. After the crimping and staple cutting operations the fiber was opened a 12-inch (30.5 cm) wide microdenier metallic card in lots of 30 grams. The open fiber was then used to produce a batt on a sample card line. This carded batt was needled on a James Hunter Fiberlocker Needle machine to give a resultant needle-punched fabric.

Example 5

Cotton fibers (4 kg, 1.5 to 5 cm in length) and 7-denier bicomponent fibers (30/70 (w/w) poly(hydroxyaminoether)/polypropylene), (0.45 kg, 2.5 cm in length) were manually mixed and then opened up. The blend was carded and converted into a non-woven web, which was thermally bonded with calender rolls at 170°C.

Example 6

Cotton linters (150 g) and 7-denier bicomponent fibers (30/70 (w/w) polyetheramine/polypropylene) were added to 300 L of water in a cylindrical tank and the contents were agitated for 5 minutes. The polyetheramine was derived from the reaction

of the diglycidyl ether of bisphenol A and ethanolamine. The ratio of bicomponent fibers to cotton linters was 5 percent on a weight basis. The slurry was then pumped to a moving belt made of polyester mesh and the webs formed were collected. The wet webs were dried by passing them through a 165°C oven with approximately one minute of 5 dwell time. The dried webs were then bonded with heated calender rolls at temperatures from 100°C to 180°C. The basis weight of the webs after bonding was about 90 gsm.

Example 7

A bicomponent, sheath/core spunbond fabric was produced under the conditions shown in Table II. The polyetheramine (derived from the reaction of the 10 diglycidyl ether of bisphenol A and ethanolamine) sheath had a MFI of 15. The polypropylene core was composed of a 35 MFI Pro-fax™ PF635. The sheath/core ratio was 20/80 (w/w). The system was run at slot air pressures of 20, 25 and 30 psi and the spunbond material was collected on perforated belt/vacuum collection system. Collection speeds ranged from 50 to 75 meters per minute. Calender rolls were set to 60°C and 15 appeared to provide adequate bonding for dry web stock.

Table II

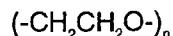
GENERAL	SHEATH EXTRUDER	CORE EXTRUDER
Polymer Type	Dow Polyetheramine (15 MFI)	Montell Polypropylene (35 MFI)
Ratio (w/w)	20	80
Extrusion Temps, C		
Zone 1	180	195
Zone 2	185	215
Zone 3	184	225
Zone 4	195	223
Melt Pump Pressure (psi)	840	1030
Meter Pump (rpm)	6.2	53.1
Forming Table Speed (meters/min)	74.8	
Top Calender Roll Spd(meters/min)	75.4	
Bott Calendar Roll Spd(meters/min)	76	
Calender Temperature (°C)	60	
Force (N/mm)	80	

20

Examples 8 Through 15 - PHAE Blends for Hydrophilic Fiber/Fabric Applications

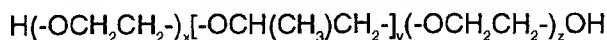
The poly(hydroxy amino ether) ("PHAE") used in the following Examples 8 through 15 was produced by The Dow Chemical Company from the polymerization of a diglycidyl ether of Bisphenol A and ethanol amine. The PHAE had the following 25 properties: number average molecular weight (Mn) = 14,000; weight average molecular weight (Mw) = 35,000; melt index = 15 (measured at 190°C with a 2.16 kg weight); glass

transition temperature (Tg) = 78°C. In these Examples 8 through 15, PEG refers to poly(ethylene glycol) and PEO refers to poly(ethylene oxide). PEG and PEO have the same polyoxyethylene repeating unit shown below:



5 Designation of polymers with the above structure as PEG or PEO was based on the product name given in an Aldrich catalogue, that is, PEG is used if the number average molecular weight (Mn) of the polyoxyethylene is 10,000 or less, and PEO if the viscosity average molecular weight (Mv) is 100,000 or above. In the following examples, the number immediately after PEG or PEO indicates the calculated average molecular weight 10 (Mn or Mv) from the Aldrich catalog.

EPE refers to a block copolymer with the general structure shown below:



15 The EPE block copolymers contain a hydrophobic block of poly(propylene oxide), having a molecular weight ranging from a minimum of 900 to a maximum of 4000, with two hydrophilic polyoxyethylene blocks such that the combined weight of the polyoxyethylene blocks constitutes from 10 to 90 weight percent of the total molecule. The EPE block copolymers are nonionic surfactants [see L. G. Lundsted and I.R. Schmolka, "The Synthesis and Properties of Block Copolymer Polyol Surfactants" in *Block and Graft Copolymerization, volume 2* (edited by R. J. Ceresa), John Wiley and Sons, New York, chapter 1, pp. 1-103]. The EPE block copolymers are marketed under 20 the Trademark PLURONIC® polyols (BASF Wyandotte Corporation) and are also sold by The Dow Chemical Co. (for example, Polyglycol EP-1730 and EP-1660).

25 The nomenclature used herein for specific EPE block copolymers gives the weight percent ethylene glycol and the average calculated molecular weight (Mn), as given in the Aldrich catalog. For example, EPE-30 (Mn 5800) refers to an EPE block copolymer containing 30 weight percent ethylene glycol and having a calculated number average molecular weight of 5800.

The following test methods were used for Examples 8 through 15

30 Glass transition temperature (Tg) was determined using a TA Instruments DSC 2010 Differential Scanning Calorimeter. Samples (5 to 10 mg) were prepared in hermetically sealed pans. Two scans were made for each sample. The first scan was made from ambient to 200°C at 10°C per minute. The sample was then cooled to ambient temperature or below using dry ice, whereupon the second scan was made at 10°/minute to 200°C. The Tg was determined from the second scan inflection point.

pH7 Buffer Contact Angle was determined for compression molded films using a Krüss G40 Contact Angle Measuring System (Goniometer) equipped with a Eurometrix fiber optic light source, a Kernco model G-1 microscope, stage, light source, and camera mount, and a Kruss Panasonic CCTV camera and WV-5410 monitor. A 5 small drop of the pH7 buffer was applied to the film and the angle formed at the film/drop/air interface was measured using the system software (G40 V1.32-US). In the examples that follow the terms "water contact angle" and "pH7 buffer contact angle" are synonymous.

10 Laboratory-scale fiber spinning of PHAE blends. The apparatus for spinning fiber consisted of a Rheometrics capillary melt rheometer equipped with a 1000 μm die, a Rheotens attachment, and a 12 inch (30.5 cm) circumference variable speed roll onto which the fiber was spun.

Example 8 - PHAE Blend With 10 Weight Percent PEG 10,000

15 PHAE (243.0 g) and PEG 10,000 (27.1 g, Aldrich Chemical Co., Tm 63°C) were melt blended for 20 minutes in a large capacity Haake Torque rheometer (with roller mixing blades) at 170°C isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 53°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 67. Fiber was melt spun at 190°C with a rheometer plunger speed of 0.3 inch/minute and a take-up roll speed of 1780 rpm (543 m/minute). Additional blends with 5 and 25 weight percent PEG 20, 10,000 were also prepared and the results are included in Table III.

Example 9 - PHAE Blend With 5 Weight Percent PEO 100,000

25 PHAE (57.1 g) and PEO 100,000 (3.1 g, Aldrich Chemical Co., Tm 65°C) were melt blended in a Haake Torque rheometer for 15 minutes at 180°C isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 66°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 68. Fiber was melt spun at 190°C with a plunger speed of 0.3 inch/minute (8mm/minute) and a take-up speed of 1780 rpm (543 m/minute). The denier of the fiber was 9 g (equals the weight of 9000 m of 30 continuous fiber). Additional blends with 10 percent and 25 percent PEO 100,000 were prepared and the results are included in Table III.

Example 10 - PHAE blend with 5 Weight Percent PEO 4,000,000

PHAE (57.0 g) and PEO 4,000,000 (3.0 g, Aldrich Chemical Co., Tm 65°C) were melt blended in a Haake torque rheometer for 20 minutes at 180°C

isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 67°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 65. Fiber was melt spun at 190°C with a plunger speed of 0.3 inch/minute (8 mm/minute) and a take-up speed of 1780 rpm (543 m/minute). The denier of the fiber was 10 g.

Example 11 - PHAE Blend With 10 Weight Percent PEO 4,000,000

10 PHAE (243.0 g) and PEO 4,000,000 (27.0 g, Aldrich Chemical Co., Tm 65°C) were melt blended in a Haake torque rheometer for 25 minutes at 180°C isothermal metal temperature and 100 rpm mixing speed. The resulting blend had a Tg of 55°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 72. Fiber was melt spun at 190°C with a plunger speed of 0.3 inch/minute (8 mm/minute) and a take-up speed of 1780 rpm (543 m/minute).

Example 12 - PHAE Blend With 15 Weight Percent PEO 4,000,000

15 PHAE (51.0 g) and PEO 4,000,000 (9.0 g, Aldrich Chemical Co., Tm 65°C) were melt blended in a Haake torque rheometer for 20 minutes at 180°C isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 44°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 42. Fiber was melt spun at 190°C with a plunger speed of 0.3 inch/minute (8 mm/minute) and a take-up speed of 1100 rpm (335 m/minute). Additional blends with 20 and 25% PEO 4,000,000 were prepared and the results are included in Table III.

Example 13 - PHAE Blend With 3.7 Weight Percent EPE-30 (Mn 5800)

20 PHAE (73.4 g) and EPE-30 (2.8 g, Aldrich Chemical Company, Tm 39°C) were melt blended in a Haake torque rheometer for 20 minutes at 180°C isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 69°C with no crystalline melting point. A compression molded film of the blend had a pearlescent, semi-transparent appearance and had a water contact angle of 24. Fiber was melt spun at 200°C with a plunger speed of 0.3 inch/minute and a take-up speed of 1780 rpm (543 m/minute). The denier of the fiber produced was 8g. A small hand sample of fabric was made from the fiber as follows: A portion of the fiber (1.4 g) was cut into 2 inch staple and carded to make a web. The web was folded in half and then run through a Beloit Wheeler Model 700 Lab Calendar Roll. The calender rolls were set at 210°F and 1000 psi which gave a well bonded fabric. A sample of pure PHAE fiber (1.4 g) was made into

fabric using a similar procedure. The ability of the fabrics to wick water was then tested by immersing one end of a small strip (19 by 81 mm) of each fabric into deionized water and measuring the time required for the water to advance up the fabric strip to a line drawn 25 mm from the surface of the water. The PHAE blend with EPE-30 (Mn 5800) 5 wicked water to the line in 2.5 minutes whereas the pure PHAE fabric showed no wicking of water at all. The fabrics were redried and the wicking test was repeated 3 times. The same results were observed each time - the fabric made with the EPE-30 blend wicked water and the pure PHAE fabric did not.

Example 14 - PHAE blend with 5 Weight Percent EPE-80 (Mn 8400)

10 PHAE (57.0 g) and EPE-80 (3.0 g, Aldrich Chemical Co., Tm 58°C) were melt blended in a Haake torque rheometer for 20 minutes at 180°C isothermal metal temperature and 100 rpm mixing rate. The resulting blend had a Tg of 64°C with no crystalline melting point. A compression molded film of the blend was transparent and had a water contact angle of 56. Fiber was melt spun at 200°C with a plunger speed of 15 0.3 inch/minute and a take-up speed of 850 rpm (259 m/minute). The denier of the fiber produced was 274g.

Blends of PHAE with other EPE block copolymers, including EPE-30 (Mn 4400); EPE-40 (Mn 2,900); and EPE-50 (Mn 1,900), were also prepared using procedures analogous to those described above. The results for these blends are listed 20 in Table IV.

Example 15 - PHAE blend with 5 Weight Percent Poly(propylene glycol) (Aldrich, Mn 3500)

PHAE (64.9 g) and poly(propylene glycol) (3.4 g) were melt blended in a Haake torque rheometer for 40 minutes at 150°C to 180°C isothermal metal temperature 25 and 30 to 100 rpm mixing rate. The initial blend appeared to be two-phase and mixing was very poor. Temperature and mixing rate were adjusted until good mixing was obtained. The resulting blend had a Tg of 76°C. A compression molded film of the blend was opaque.

Table III
PHAE Blends with Polyoxyethylene (PEG or PEO)

PHAE blend composition (a)	Tg °C	Contact angle (pH7 buffer) (b)	Comment (c)
pure PHAE	78	89 ± 5	
5% PEG 10,000	64	68 ± 4	miscible
10% PEG 10,000	53	67 ± 3	"
25% PEG 10,000	20	23 ± 5	"
5% PEO 100,000	66	68 ± 6	miscible
10% PEO 100,000	54	64 ± 4	"
25% PEO 100,000	21	25 ± 8	"
5% PEO 4,000,000	67	65 ± 4	miscible
10% PEO 4,000,000	55	67 ± 3	"
15% PEO 4,000,000	44	42 ± 6	"
20% PEO 4,000,000	37	18 ± 4	"
25% PEO 4,000,000	21	17 ± 3	"

5 a) Weight percent of additive given with the balance being PHAE.

b) Contact angle measurement for drops of pH 7 buffer solution applied to the surface of a compression molded film. Value reported is the average of results for 15 drops.

c) Samples were classified as miscible if the Tg satisfied the Fox equation and if films of the blends were transparent.

Table IV
PHAE blends with EPE Block Copolymers

PHAE blend composition (a)	Tg, °C	Contact angle (pH7 buffer) (b)	Comment (c)
Pure PHAE	78	89 ± 5	Fiber made at high line speed: denier = 7, Fabric sample does not wick water
3.7% EPE-30 (Mn 5,800)	69	24 ± 2	Immiscible blend; Fiber made at high speed: diameter = 30µm; Fabric sample exhibits wicking of water
2% EPE-30 (Mn 4,400)	78	46 ± 5	Immiscible blend
4% EPE-30 (Mn 4,400)	76	37 ± 2	"
2% EPE-40 (Mn 2,900)	75	31 ± 4	Immiscible blend
4% EPE-40 (Mn 2,900)	73	24 ± 3	"
2% EPE-50 (Mn 1,900)	73	73 ± 3	Miscible blend
5% EPE-50 (Mn 1,900)	65	34 ± 5	"
5% EPE-80 (Mn 8,400)	64	56 ± 4	Miscible blend; Fiber made at medium speed
10% EPE-80 (Mn 8,400)	55	56 ± 3	"

a) Weight percent of additive given with the balance being PHAE.

5 b) Contact angle measurement for drops of pH 7 buffer solution applied to the surface of a compression molded film. Value reported is the average of results for 15 drops.

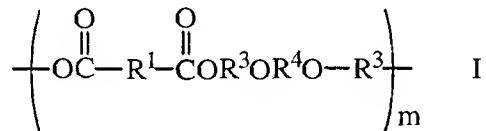
c) Samples were classified as miscible if the Tg satisfied the Fox equation and if films of the blends were transparent.

CLAIMS:

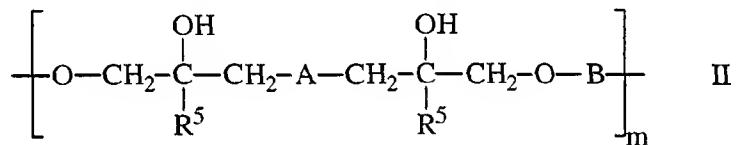
1. A fiber comprising at least one thermoplastic hydroxy-functionalized polyether or polyester.
2. The fiber of Claim 1 wherein the thermoplastic hydroxy-functionalized polyether or polyester is prepared by the reaction of a dinucleophilic monomer with a diglycidyl ether, a diglycidyl ester or epihalohydrin.

5 3. The fiber of Claim 1 wherein the hydroxy-functionalized polyether or polyester is selected from:

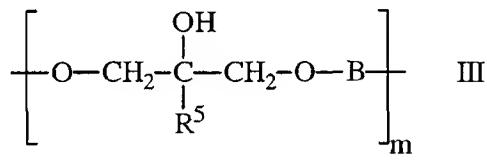
10 (1) poly(hydroxy ethers) or poly(hydroxy esters) having repeating units represented by the formula:



(2) polyetheramines having repeating units represented by the formula:

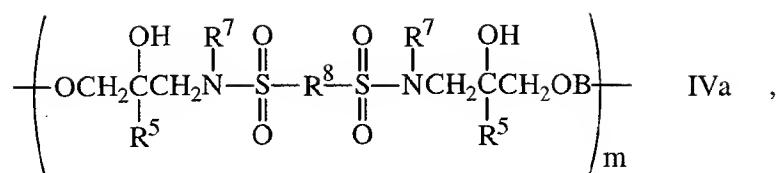


15 (3) hydroxy-functionalized polyethers having repeating units represented by the formula:

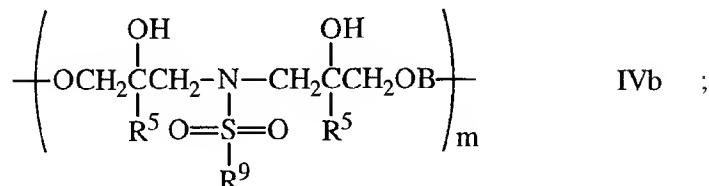


or

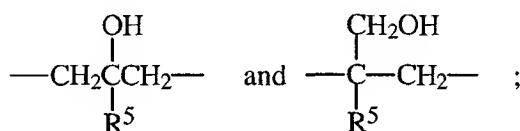
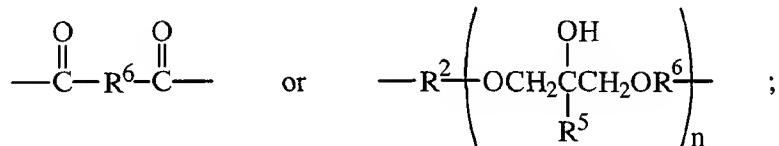
(4) hydroxy-functionalized poly(ether sulfonamides) having repeating units represented by the formula:



or



wherein R^1 is a divalent organic moiety which is primarily hydrocarbon; R^2 is independently a divalent organic moiety which is primarily hydrocarbon; R^3 is

5 R^4 is

R^5 is hydrogen or alkyl; R^6 is a divalent organic moiety which is primarily hydrocarbon; R^7 and R^9 are independently alkyl, substituted alkyl, aryl, substituted aryl; R^8 is a divalent organic moiety which is primarily hydrocarbon; A is an amine moiety or a combination of 10 different amine moieties; B is a divalent organic moiety which is primarily hydrocarbon; m is an integer from 5 to 1000; and n is an integer from 0 to 100.

4. The fiber of Claim 3 having a cylindrical, cross-shaped, trilobal or ribbon-like cross-section.

5. The fiber of Claim 3 formed by melt-spinning, dry-spinning or wet-spinning of a polymer solution.

6. The fiber of Claim 3 in the form of a filtration medium, a binder fiber for glass or carbon fibers, a binder fiber in non-woven fabrics made of a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester or a binder fiber in non-woven fabrics made of cellulosic-based materials, or a medical apparel.

7. A woven or non-woven fabric comprising the fiber of Claim 3 and, optionally, a synthetic or natural fiber.

8. The fabric of Claim 7 wherein the synthetic fiber is a polyester, a polyamide, rayon or a polyolefin and the natural fiber is cotton.

5 9. The fabric of Claim 7 in the form of an apparel, a water-absorbent cloth, a filter fabric, a battery separator, an antistatic wipe or a water-absorbent mat.

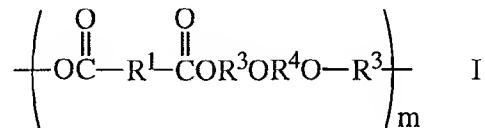
10 10. The fiber of Claim 1 comprising a blend of one or more hydroxy-functionalized polyether or polyester with a thermoplastic polymer which is not an hydroxy-functionalized polyether or polyester selected from a polyolefin, polyester, polyamide, polysaccharide, modified polysaccharide or naturally-occurring fiber or particulate filler, thermoplastic polyurethane, thermoplastic elastomer or glycol-modified copolyester (PETG).

15 11. The fiber of Claim 1 which is a bicomponent fiber having (1) a first component comprising a thermoplastic hydroxy-functionalized polyether or polyester or a blend of a hydroxy-functionalized polyether or polyester and (2) a second component comprising a polyolefin, polyester, polyamide, polysaccharide, modified polysaccharide or naturally-occurring fiber or particulate filler, thermoplastic polyurethane, thermoplastic elastomer or glycol-modified copolyester (PETG).

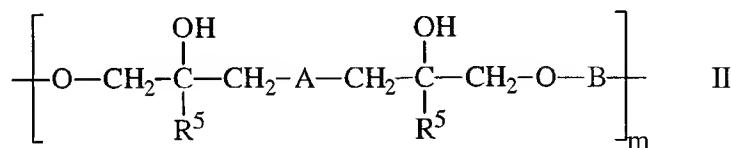
20 12. The bicomponent fiber of Claim 11 wherein the hydroxy-functionalized polyether or polyester is prepared by the reaction of a dinucleophilic monomer with a diglycidyl ether, a diglycidyl ester or epihalohydrin.

13. The bicomponent fiber of Claim 11 wherein the hydroxy-functional polyether is selected from:

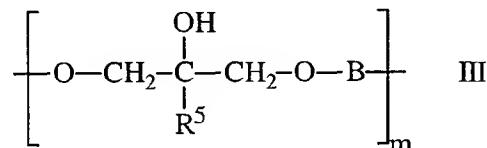
25 (1) poly(hydroxy ethers) or poly(hydroxy esters) having repeating units represented by the formula:



(2) polyetheramines having repeating units represented by the formula:



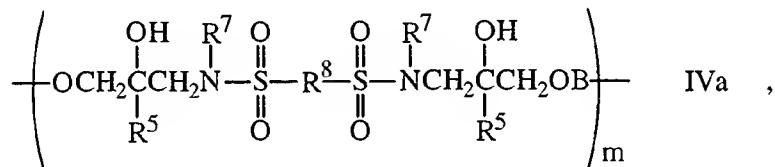
(3) hydroxy-functionalized polyethers having repeating units represented by the formula:



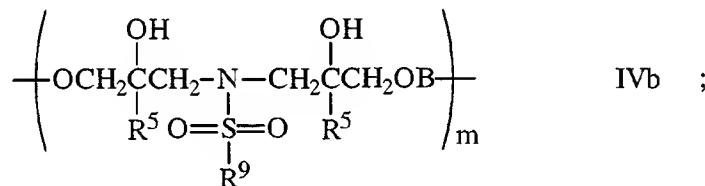
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or

(4) hydroxy-functionalized poly(ether sulfonamides) having repeating units represented by the formula:

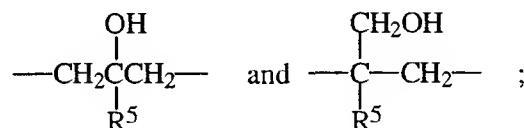


or

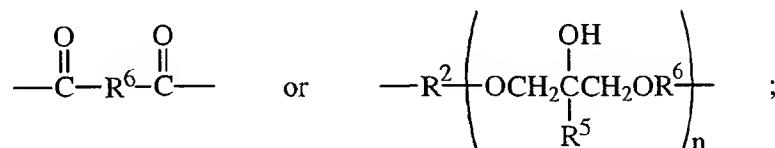


wherein R¹ is a divalent organic moiety which is primarily hydrocarbon; R² is independently a divalent organic moiety which is primarily hydrocarbon; R³ is

10



R⁴ is



R⁵ is hydrogen or alkyl; R⁶ is a divalent organic moiety which is primarily hydrocarbon; R⁷

and R⁹ are independently alkyl, substituted alkyl, aryl, substituted aryl; R⁸ is a divalent organic moiety which is primarily hydrocarbon; A is an amine moiety or a combination of different amine moieties; B is a divalent organic moiety which is primarily hydrocarbon; m is an integer from 5 to 1000; and n is an integer from 0 to 100.

5 14. The bicomponent fiber of Claim 13 which is a side-by-side bicomponent fiber, a sheath-core bicomponent fiber, a segmented-pie bicomponent fiber or an islands-in-the-sea bicomponent fiber.

10 15. The bicomponent fiber of Claim 14 comprising a core of the thermoplastic hydroxy-functionalized polyether or polyester and a sheath of a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

16. The bicomponent fiber of Claim 14 comprising a sheath of the thermoplastic hydroxy-functionalized polyether or polyester and a core of a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester.

15 17. The bicomponent fiber of Claim 13 having a cylindrical, cross-shaped, trilobal or ribbon-like cross-section.

20 18. The bicomponent fiber of Claim 13 in the form of a filtration medium, a binder fiber for glass or carbon fibers, a binder fiber in non-woven fabrics made of a thermoplastic polymer which is not a hydroxy-functionalized polyether or polyester or a binder fiber in non-woven fabrics made of cellulosic-based materials, or a medical apparel.

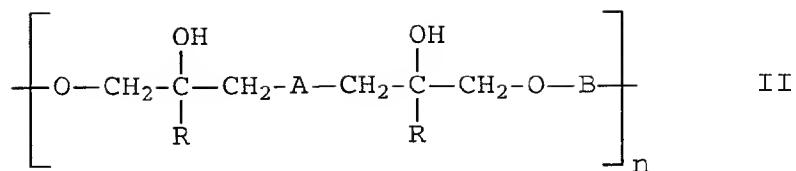
19. A woven or non-woven fabric comprising the fiber of Claim 13 and, optionally, a synthetic or natural fiber.

20 20. The fabric of Claim 19 wherein the synthetic fiber is a polyester, a polyamide, rayon or a polyolefin and the natural fiber is cotton.

25 21. The fabric of Claim 19 in the form of an apparel, a water-absorbent cloth, a filter fabric, a battery separator, an antistatic wipe or a water-absorbent mat.

22. The fiber of Claim 1 comprising a blend of:

(a) a poly(hydroxy amino ether) having repeating units represented by the formula:



wherein A is a diamino moiety or a combination of different amine moieties; B is a divalent organic moiety which is predominantly hydrocarbylene; R is alkyl or hydrogen; and n is an integer from 5 to 1000; and

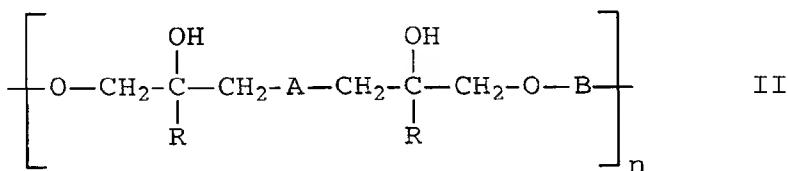
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(b) at least one of a polyethylene glycol, poly(ethylene oxide), or EPE block copolymer.

23. The fiber of Claim 22 wherein the poly(hydroxy amino ether) is the reaction product of a diglycidyl ether of bisphenol-A and ethanolamine.

10 24. A method of forming a non-woven fabric by forming a web of at least one fibrous component and heating the web to cause bonding of fibrous components of the web, characterized in that at least one fibrous component comprises a thermoplastic hydroxy-functionalized polyether or polyester.

15 25. The method of Claim 24 wherein at least one fibrous component comprises a poly(hydroxy amino ether) having repeating units represented by the formula:



20 wherein A is a diamino moiety or a combination of different amine moieties; B is a divalent organic moiety which is predominantly hydrocarbylene; R is alkyl or hydrogen; and n is an integer from 5 to 1000.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/28462

A. CLASSIFICATION OF SUBJECT MATTER	IPC 7	D01F6/66	D01F6/84	D01F6/86	D01F8/14	D01F8/16
		D04H1/54				

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC 7 D01F D04H

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 98 51728 A (BECKERDITE JOHN M ;WHITE JERRY E (US); DOW CHEMICAL CO (US); MANG) 19 November 1998 (1998-11-19) page 1, line 17 - line 28	1-5
Y	page 5, line 32 -page 8, line 29; claims ---	6-14, 16-21,24
X	EP 0 780 438 A (BASF AG) 25 June 1997 (1997-06-25) the whole document ---	1-5
X	US 4 337 330 A (ROBESON LLOYD M) 29 June 1982 (1982-06-29) column 1, line 37 - line 47 column 2, line 50 -column 6, line 27; claims ---	1-5
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Further documents are listed in the continuation of box C.

Patent family members are listed in annex.

Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance
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 "P" document published prior to the international filing date but later than the priority date claimed

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Date of the actual completion of the international search	Date of mailing of the international search report
20 March 2000	29/03/2000
Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+31-70) 340-3016	Tarrida Torrell, J

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 99/28462

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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X	PATENT ABSTRACTS OF JAPAN vol. 1998, no. 03, 27 February 1998 (1998-02-27) & JP 09 302522 A (TORAY IND INC), 25 November 1997 (1997-11-25) abstract ---	1-5
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Int'l. at. J. Application No

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